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"RAPID HYDROMETER SEDIMENTATION METHOD FOR PARTICLE SIZE DISTRIBUTION ANALYSIS OF DRILLING FLUIDS."

A THESIS

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ABSTRACT

A theoretical and experimental investigation has been conducted in order to establish a rapid method of particle size distribution analysis for drilling fluids in the 2 to 74 micron range.

It was found that a modified hydrometer sedimentation method of analysis, described herein, can be most easily and inexpensively adopted for the above mentioned size interval. The method differs from the published methods mainly in the manner in which the sample is prepared for analysis, in the nonuse of a deflocculating agent, in measuring the viscosity of the suspension, in the time required for measuring the specific gravity of suspension, and in the calculation of the results through the utilization of prepared graphs.

The results obtained by the sedimentation method are in good agreement with those obtained by microscopic and sieve analyses. The development and experimental verification of the method are presented and its reliability and relative advantages and disadvantages are discussed.

ACKNOWLEDGEMENTS

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INTRODUCTION

When clays are mixed with water to make mud fluids for use in drilling wells for oil and gas the liquid mixtures have certain physical characteristics that vary with the quantity and the size of their inherent solid particles. A knowledge of particle size distribution is essential for a better understanding of fundamental properties of drilling fluids, particularly viscosity and gel strength as Jessen and Mungan (1) pointed out. Furthermore, particle size analysis can give valuable information for the classification of muds and injection waters for formation damage studies, and for deciding whether it is advantageous or not to operate special equipment for particle size control in the development drilling and subsequent operation of an oil field.

Although several methods are available for particle size distribution analysis (2), there are only a limited number of them that can be adopted for the study of drilling fluids. Particles larger than 38 microns (400 mesh) may be obtained and classified by wet screening. For sizes between 38 and 2 microns the analysis may be carried out by sedimentation, light microscopy or turbidimetry. Below the 2 micron size, electronmicroscopy or supercentrifuge methods may be employed.

The primary purpose of this investigation was to establish a rapid method of particle size distribution analysis for the 2 to 74 micron range. At the outset of the project two methods were considered, namely, light microscopy and sedimentation. It was found that the hydrometer sedimentation method of analysis can be most easily and inexpensively adopted for the study of

drilling fluids in the above mentioned size interval. The light microscopic method of analysis described by Fairs (3) proved to be too tedious and time consuming. Therefore, it was abandoned in the early stage of the investigation as a rapid means of analysis and was employed only for checking the results obtained by the sedimentation method.

In 1927 Bouyoucos (4) described a hydrometer method for determining the colloid material of soils, which was modified slightly by Bauer (5) in 1937. Following some changes in 1938, the method was adopted as A.S.T.M. Standard in 1939. Two revisisions that were more or less editorial in nature were included in the Standard in 1951. An extensive revision was made in 1954 and was described by Bauer (6).

The method described herein differs from the published methods mainly in the manner in which the sample is prepared for analysis, in the nonuse of a deflocculating agent, in measuring the viscosity of the suspension, in the time required for measuring the specific gravity of suspension, and in the calculation of the results through the utilization of prepared graphs.

THEORY OF SEDIMENTATION

A solid particle falling in a medium of lesser density rapidly accelerates to a terminal velocity, descending thereafter at this constant rate, determined, for a particular medium by the size and density of particle. There exist three distinct modes of fall - the viscous, transition and turbulent. The limits of each of these regions are determined by the Reynold's number which is related to the velocity of the particle with respect to the medium, to the size of the particle and to the viscosity and density of the medium as follows:

$$\text{Re.No.} = \frac{D\ v\ d_1}{n_1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

where:

Re.No. = Reynold's number, dimensionless

D = Diameter of particle, cm.

v = Terminal velocity of particle, cm/sec.

d_1 = Density of the medium, gm/ml.

n_1 = Viscosity of the medium, poises.

Thus, the diameter limits of the various sedimentation regions are not absolutely defined but vary with the material and medium employed.

The turbulent region is defined by Davis (7) as involving Reynold's numbers of the order of 80 and larger, the transition zone

prevails between Reynold's numbers of 80 and 0.5 and viscous or laminar conditions exist at Reynold's numbers less than 0.5.

Stokes (8) showed that the fall of spherical particles of nonporous, incompressible solid in an incompressible fluid in laminar flow is governed by the following law:

$$D' = \sqrt{\frac{18\, n_1^* v}{980\ (d_s - d_1)}} \qquad \text{..... (2)}$$

where:

D' = particle diameter, cm.

n_1^* = viscosity of the medium, poises.

v = velocity of particle, cm/sec.

d_s = density of solids, gm/ml.

d_1 = density of the medium, gm/ml.

For particle size distribution calculations Stokes' law may be transformed to a more convenient form such as:

$$D = 17.5 \sqrt{\frac{n_1}{d_s - d_1}} \sqrt{\frac{h}{t}} \qquad \text{..... (3)}$$

where:

D = particle diameter, microns

n_1 = viscosity of the medium, centipoises.

d_s = density of solids, gm/ml.

d_1 = density of the medium, gm/ml.

h = effective depth, cm.

t = settling time, min.

Combining equation (3) with equation (1) and taking the Reynold's number as 0.5, the upper limit of particles can be calculated that settle according to Stokes' law, as follows:

$$D_c = 92.2 \left[\frac{n_1^2}{d_1 (d_s - d_1)} \right]^{1/3} \quad \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

where:

D_c = critical diameter, microns.

n_1, d_s and d_1 as defined previously.

This diameter is usually called critical diameter.

On the other hand, according to Bray (9) the smallest particles that settle from suspension in accordance with Stokes' law are about 0.5 micron in equivalent spherical diameter. Deviations from Stokes' law develop when the particle diameter is only slightly larger than the free mean path of the fluid molecules. Under such conditions the mass of the solid particles is small enough to be affected by the Brownian movement of the suspending medium, which compels the particles to remain in suspension in opposition to the force of gravity.

THE HYDROMETER SEDIMENTATION METHOD OF ANALYSIS

In principle the hydrometer method of particle size analysis consists of sedimentation of particles with direct physical measurement of the specific gravity of the suspension as a function of time. The rate of settling of particles in the suspension is computed from measurements of the specific gravity of the suspension observed at measured depths by means of a hydrometer at definite time intervals. The spherical diameter of those particles that have just reached a measured depth after a definite time interval can be calculated from equation (3), and by calculating the density of the suspension at the measured depth the weight percent of particles remaining in that depth can be computed as follows:

$$W = \frac{1000}{X} \quad \frac{d_s}{d_s - d_l} \quad (d_m - d_l) \times 100\% \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (5)$$

where:

W = weight percent smaller than corresponding size, D, percent.

X = weight of solid materials per 1000 ml of suspension, gm.

d_s = density of solids, gm/ml.

d_l = density of the medium, gm/ml.

d_m = density of suspension, gm/ml.

The effective depth, h, appearing in Stokes' law must be evaluated from each specific gravity observation in order

to calculate the particle diameters.

The relationship between the effective depth and the hydrometer readings may be derived from the following considerations*:

The observed specific gravity is the specific gravity of the suspension at the volume center of the submerged portion of the hydrometer. The distance from the surface of the suspension to the volume center of the hydrometer is the effective depth, h, and, if the volume of the submerged part of the hydrometer stem is neglected, see Fig. 1, then

$$h = L_1 + 1/2 \left(L_2 - V_B/A \right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (6)$$

where:

h = effective depth, cm.

L_1 = distance from top of suspension to top of hydrometer bulb, cm.

L_2 = length of hydrometer bulb, cm.

V_B = volume of bulb, cm^3.

A = cross-sectional area of cylinder containing suspension, cm^2.

Once the values of the above mentioned terms have been determined, the relationship between h and the hydrometer readings can be calculated.

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* The calculation of effective depth is based on the method described by Bauer in Reference (5).

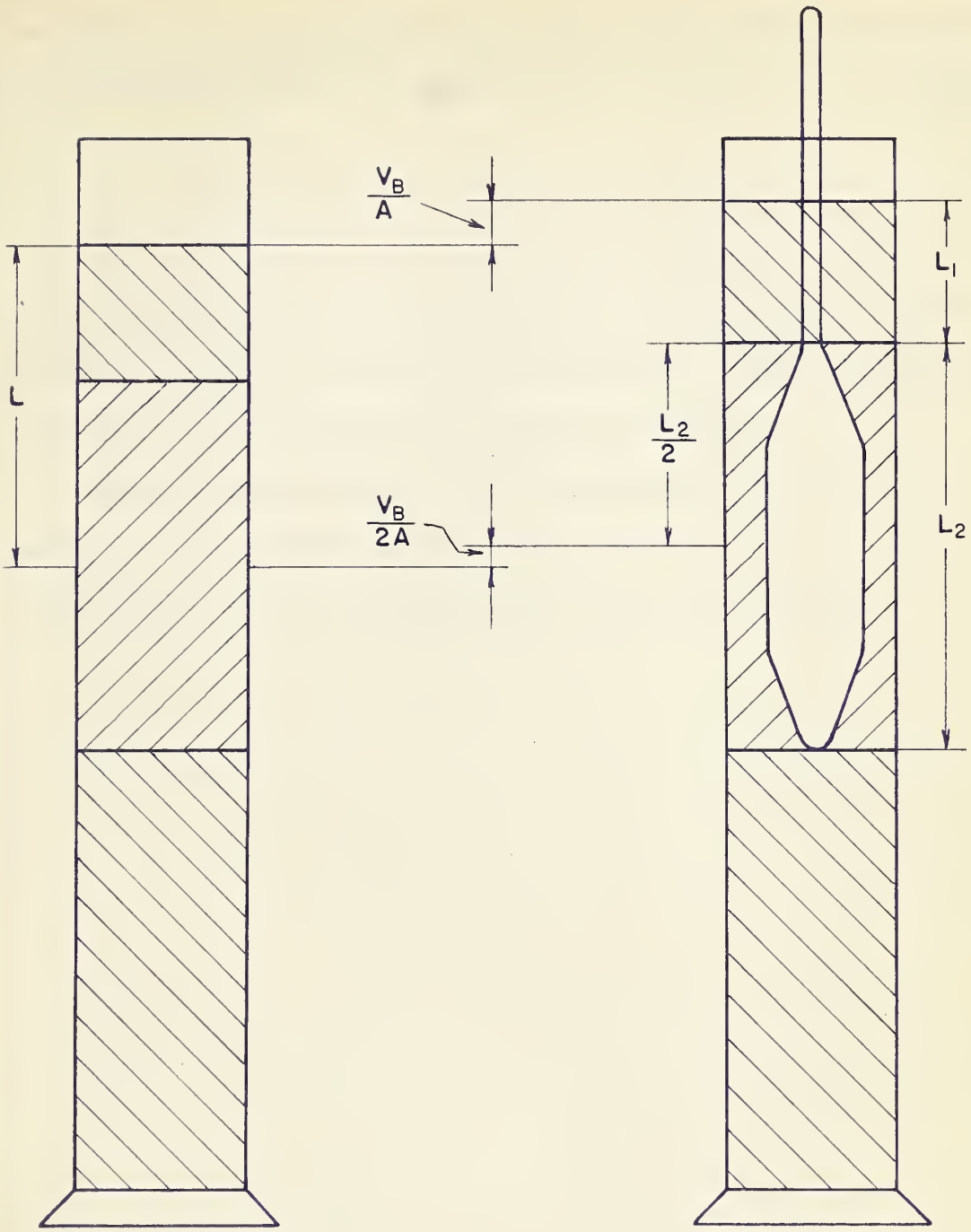

FIGURE 1. RELATIONS FOR COMPUTING EFFECTIVE DEPTH FORMULA.

It is apparent from the foregoing argument that the effective depth, h, is a linear function of the specific gravity of the suspension, so the equation for h reduces to a simple form

$$h = a - bG \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (7)$$

where a and b are constants and G is the specific gravity of the suspension plus the meniscus correction. (The evaluation of these constants will be given in a later section.)

Thus, measuring the specific gravity of the suspension at definite time intervals and with the help of equations 3, 5 and 7 the particle size distribution can be determined.

MODIFIED HYDROMETER SEDIMENTATION METHOD OF ANALYSIS

The shortcoming of the previous method is the length of time required to obtain the distribution function. For instance, to gain information about 2 micron particles generally 24 hours settling time is required. To eliminate these time consuming measurements and to speed up the analysis, a modified method has been developed.

This method involves three specific gravity measurements, after settling times of 3, 10 and 30 minutes, and the use of three prepared graphs based on equations 3, 5 and 7, which for the sake of argument are repeated here:

$$D = 17.5\sqrt{\frac{n_1}{d_s - d_1}}\ \sqrt{\frac{h}{t}} \qquad \text{.....(3)}$$

$$W = \frac{1000}{X}\ \frac{d_s}{d_s - d_1}\ (d_m - d_1) \times 100\% \qquad \text{.....(5)}$$

$$h = a - bG \qquad \text{.....(7)}$$

It is evident from equation (3) that by measuring n_1, d_s and d_1, the particle size, D, is a function of the square root of particle velocity, h/t, only. Equation (3) then simplifies to

$$D = K\sqrt{\frac{h}{t}} \qquad \text{.....(3a)}$$

By taking the logarithm of both sides, it follows that

$$\log D = \log K + 1/2 \log \frac{h}{t} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3b)$$

which is a straight line on log-log coordinates, having a slope of 0.5 and an intercept of K at $\frac{h}{t} = 1.0$, as shown in Fig.2.

From the D vs. $\frac{h}{t}$ relationship $\frac{h}{t}$ can be obtained for any D, but the absolute values of h and t must be obtained by establishing a relationship between $\frac{h}{t}$ and t. This relationship is also plotted on Fig. 2, giving a composite graph.

$$\text{Calling } \frac{h}{t} = v \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (8)$$

where:

v = particle velocity, cm/min.

h = effective depth, cm.

t = settling time, min.

and taking the logarithm of both sides, gives

$$\log v = \log h - \log t \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (8a)$$

from which it can be seen that if the effective depth, h, were constant, then the relationship between v and t would be a straight line with a slope of minus one and an intercept of h at t = 1.0.

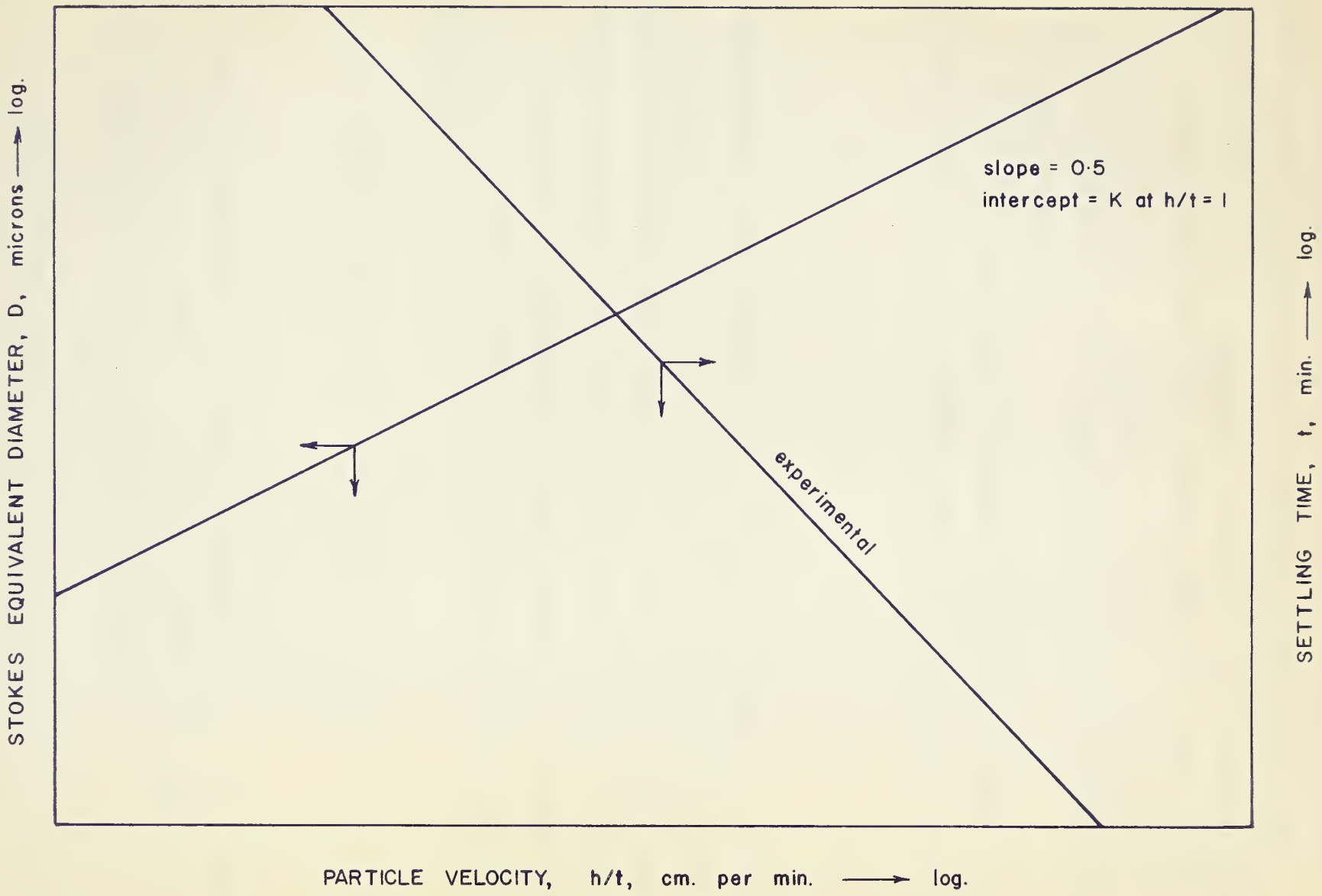

FIG. 2. PARTICLE DIAMETER AND SETTLING TIME AS A FUNCTION OF PARTICLE VELOCITY.

However, this is not the case in the hydrometer sedimentation method, because h is continually changing with time. Nevertheless, it has been found experimentally that the straight line relationship does exist between v and t but with a slope different than unity. Therefore, the $\frac{h}{t}$ vs. t line must be determined from experimental measurements of the specific gravity as a function of time.

As has been mentioned previously the effective depth is a linear function of the specific gravity of the suspension, so

$$h = a - bG \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (7)$$

where a and b are constants for a particular hydrometer. Thus, by assuming two values of G (e.g. 1.002 and 1.0100) and calculating the corresponding values of h, the straight line relationship between h and G can be established as shown in Fig. 3.

The problem of finding the weight percent, W, corresponding to a D in question is also solved with the aid of a prepared graph. Referring to equation (5)

$$W = \frac{1000}{X} \; \frac{d_s}{d_s - d_1} (d_m - d_1) \times 100\% \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (5)$$

it can be seen that W is a linear function of $(d_m - d_1)$, since d_s, d_1 and X have constant values for a particular sample, therefore, equation (5) may be reduced to the form of

$$W = K^1 (d_m - d_1) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (5a)$$

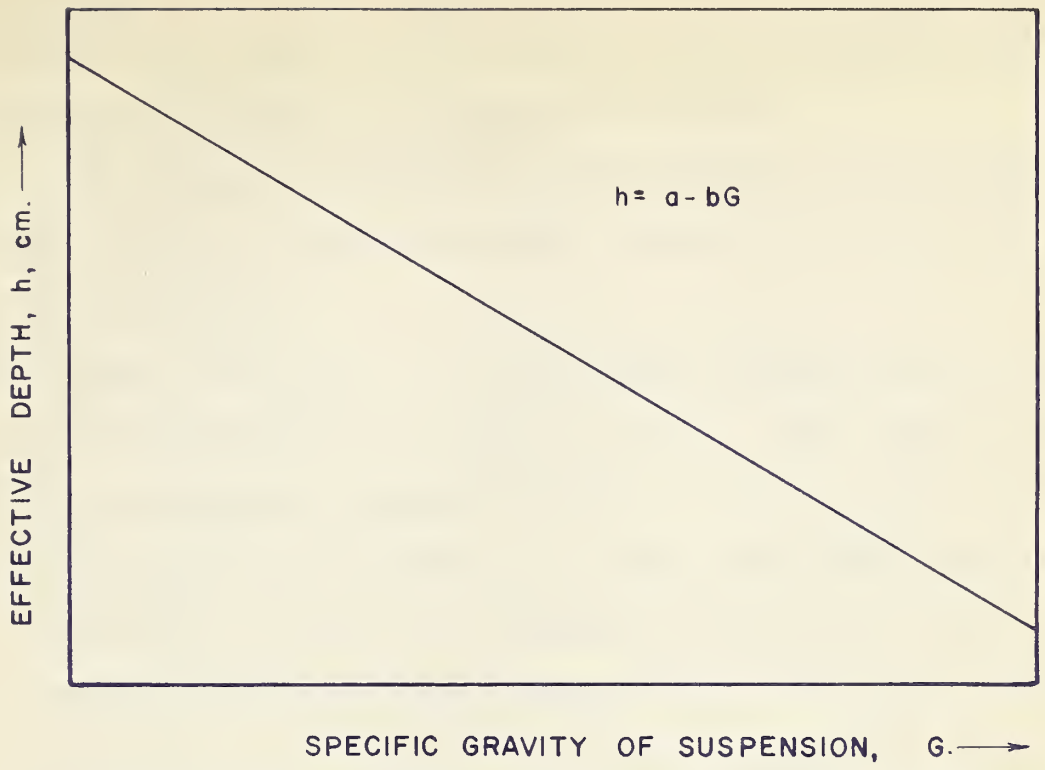

FIG. 3. EFFECTIVE DEPTH vs. SPECIFIC GRAVITY OF SUSPENSION.

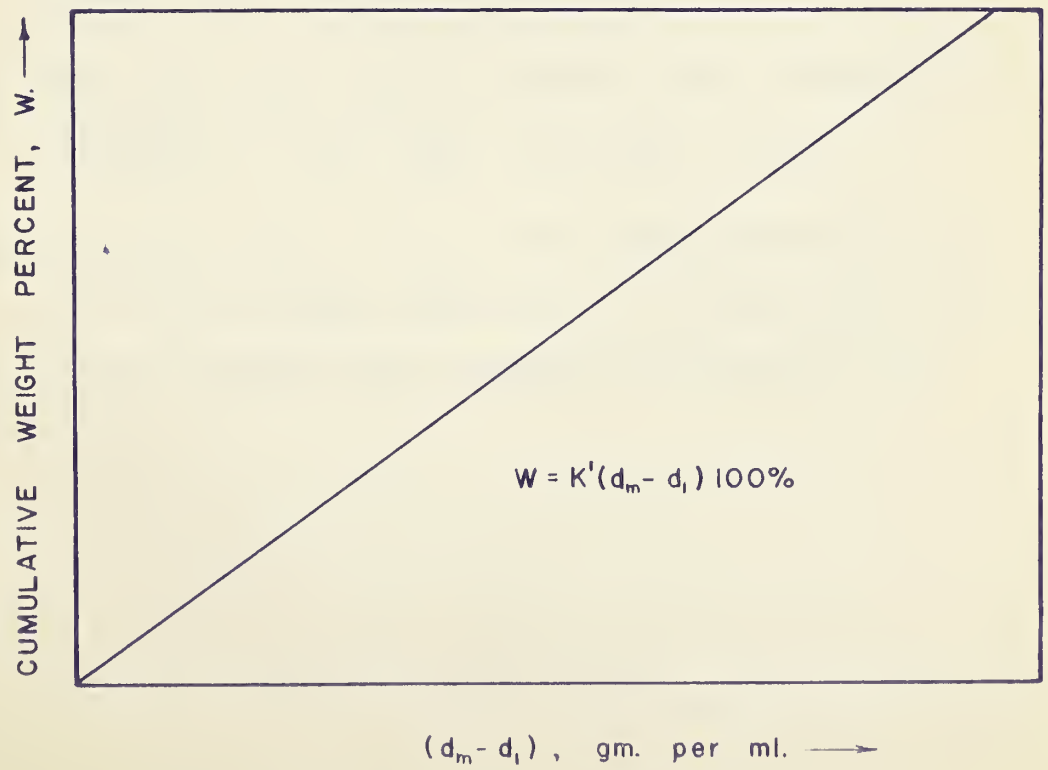

FIG. 4. CUMULATIVE WEIGHT PERCENT vs. $(d_m - d_1)$.

Plotting W vs. $(d_m - d_1)$ on linear coordinates a straight line results, the slope of which depends on d_s, d_1 and X, and passes through the origin as illustrated on Fig.4.

The method of finding the distribution function for a sample with the aid of the three graphs, similar to Figures 2, 3 and 4, is as follows:

1. Choose any time, t, on the time ordinate, Fig. 2, and proceed horizontally to the $\frac{h}{t}$ vs. t line, then at constant $\frac{h}{t}$ (vertically) proceed to the D vs. $\frac{h}{t}$ line and read the corresponding diameter, D. This value gives the Stokes equivalent diameter of particles remaining in the suspension at the effective depth measured after the time in question.

2. Find h by multiplying $\frac{h}{t}$ by t.

3. Obtain G from the h vs. G relationship, Fig. 3.

4. Calculate d_m by multiplying G by the density of distilled water, d_1, at the test temperature.

5. Find the weight percent smaller than corresponding size, W, from the W vs. $(d_m - d_1)$ chart, Fig.4.

6. Finally, plot D vs. W on semilogarithmic paper, plotting W on the linear scale and D on the logarithmic scale and draw a smooth curve through the obtained points.

APPARATUS

The apparatus for measuring the particle size distribution of drilling fluids consists of

1. Analytical balance sensitive to 1 milligram for a load of 200 grams.
2. 125 milliliter evaporating dishes.
3. Pycnometer or specific gravity flask for determining the density of solid materials.
4. Mixing apparatus.
5. 1000 milliliter glass cylinders, the inside diameter of which shall be such that the 1000 ml. mark is 36 ± 2 cm from the bottom on the inside.
6. Specific gravity hydrometer, having a range of 0.9950 to 1.0400 graduated in 0.001 specific gravity divisions.
7. Constant temperature water bath.
8. Rotating cylinder type viscosimeter (Fann V.G. meter).
9. Series of sieves numbered U.S. Standard 30, 50, 70, 100, 140, 200.

EXPERIMENTAL PROCEDURE

1. Calibration of hydrometer.

In order to find the effective depth corresponding to a specific gravity observation, the hydrometer must be calibrated. Previously it was shown, that

$$h = L_1 + 1/2 \left(L_2 - V_B/_A\right) \quad \text{.....(7)}$$

where:

V_B, the volume of the hydrometer bulb, is measured by displacement of water in a graduated cylinder in cubic centimeters.

A, the cross-sectional area of the cylinder containing suspension, is calculated from the measured inside diameter of the cylinder in square centimeters.

L_2, the length of the hydrometer bulb, is measured by calipers and a centimeter scale in centimeters.

L_1, is the measured distance from the top of the hydrometer bulb to the bottom of the specific gravity scale plus the length of the submerged portion of the specific gravity scale, to be determined, in terms of the specific gravity of the suspension.

Example of determining the effective depth formula of a hydrometer.

Specific gravity range of the hydrometer..... 0.9950 - 1.0400

Length of specific gravity scale .... 12.10 cm.

Distance from top of bulb to 1.0400 mark .... 1.20 cm.

Length of hydrometer bulb, L_2.................. 13.60 cm.

Volume of hydrometer bulb, V_B .... 45.00 $cm.^3$

Cross-sectional area of cylinder, A............. 26.41 $cm.^2$

$$L_1 = 1.20 + \frac{12.10}{1.0400 - 0.9950}(1.0400 - G)$$

$$L_1 = 280.85 - 268.89\ G$$

where G is the specific gravity of the suspension plus the meniscus correction (+0.0003) as a result of the surface tension of the suspension.

By equation (7) for Hydrometer No. 1.

$$h = 280.85 - 268.89\ G + 1/2\left[13.60 - \frac{45.00}{26.41}\right]$$

$$h = 286.76 - 268.89\ G \qquad (8)$$

2. Determination of the density of solid materials.

The density of the solid materials may be determined with the aid of a calibrated pycnometer, the volume of which is known at least to three decimal places, and a controlled temperature water bath.

The empty pycnometer is weighed, about 10 grams of oven dry ($230^{o}F$) sample placed in it and weighed again. The pycnometer then is filled with kerosene and after taking care so that no air bubbles are trapped in the sample, the pycnometer is placed in the water bath. The pycnometer and its contents are allowed to attain a temperature at which the density of the kerosene is known to four decimal places. When this temperature is reached (about 15 minutes) the pycnometer is withdrawn from the bath, carefully wiped dry and weighed.

The density is determined from

$$d_s = \frac{W_s - W_f}{V_c - V_l} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (9)$$

where:

d_s = density of solids, gm/ml.

W_s = weight of pycnometer with sample, gm.

W_f = weight of empty pycnometer, gm.

V_c = Total volume of pycnometer (known from calibration), ml.

$V_l = \frac{W_t - W_s}{d_l}$ = volume of liquid in pycnometer, ml.

W_t = weight of pycnometer with sample and kerosene, gm.

d_k = density of kerosene, gm/ml.

An alternate method may also be used instead of the one described above, which makes use of a flask with a graduated neck of which the volume change can be directly read after adding a preweighed amount of sample (10 gms.) to the kerosene. The density is calculated from

$$d_s = \frac{\text{weight of sample, gm.}}{\text{volume change of kerosene, ml.}}$$

For routine checks this method is satisfactory, but where greater accuracy is required it is not recommended by the author.

3. Preparation of sample and sedimentation test.

Fifty milliliter of the fluid sample is evaporated to dryness at 230°F in order to determine the solid content. After determining the quantity of oven-dry material per unit volume of fluid a sample containing the equivalent of 30 gms of solids is taken for the test.

The sample is placed in a dispersion cup, about 300 ml of distilled water is added and then the contents of the cup are thoroughly mixed by means of a stirring apparatus for about 5 minutes, after which the mixture is washed into a 1000 ml. cylinder and distilled water is added until the liquid level reaches the 1000 ml. mark. The cylinder then is placed in the constant temperature bath, and when the suspension attains the temperature

of the bath the cylinder is withdrawn from the bath, thoroughly shaken (using the palm of the hand to cover the mouth of the cylinder), and then replaced in the bath and the time is noted.

The specific gravity of the suspension is determined by means of a hydrometer at time intervals of 3, 10 and 30 minutes. About 20 seconds before each reading the hydrometer is lowered slowly and carefully into the suspension to minimize turbulence. The specific gravity is read on the hydrometer scale immediately after it comes to rest. The hydrometer is not permitted to remain in the suspension since particles of the suspended material settle on its shoulders and affect its accuracy. After removal, the hydrometer is wiped clean and immersed in a cylinder containing distilled water in the constant temperature bath.

After the 30 minutes reading the viscosity of the suspension is determined by means of a calibrated Fann V.G. meter.
Finally, all of the suspension is washed on a 200 mesh sieve, and the material retained on the screen is dried and then analyzed for particle size distribution by sieving.

CALCULATION AND GRAPHICAL REPRESENTATION

The following example demonstrates the method of calculating the results of the analysis of the particle size distribution and the simplicity of the calculations. To demonstrate in detail the calculations required in determining the particle size distribution of a drilling fluid, sample 7-13 has been selected. As has been shown previously, the calculation involves the use of equations 3, 5 and 7.

The physical properties of sample 7-13 are shown below

d_s = 2.4940 gm/ml.
d_l = 0.9982 gm/ml.
n = 1.30 cp.
T = 80°F.
C = 3% by weight

Using these values the working charts can be prepared as follows:

$$D = 17.5 \sqrt{\frac{1.30}{2.4940 - 0.9982}} \sqrt{\frac{h}{t}}$$

$$D = 16.62 \sqrt{\frac{h}{t}}$$

Thus, on log-log coordinates D vs. $\frac{h}{t}$ results in a straight line, having a slope of 0.5 and an intercept of 16.62 at $\frac{h}{t} = 1.0$ as shown on Fig.5.

From the three specific gravity measurements, 1.0140, 1.0128 and 1.0113 at times of 3, 10 and 30 minutes, respectively, the

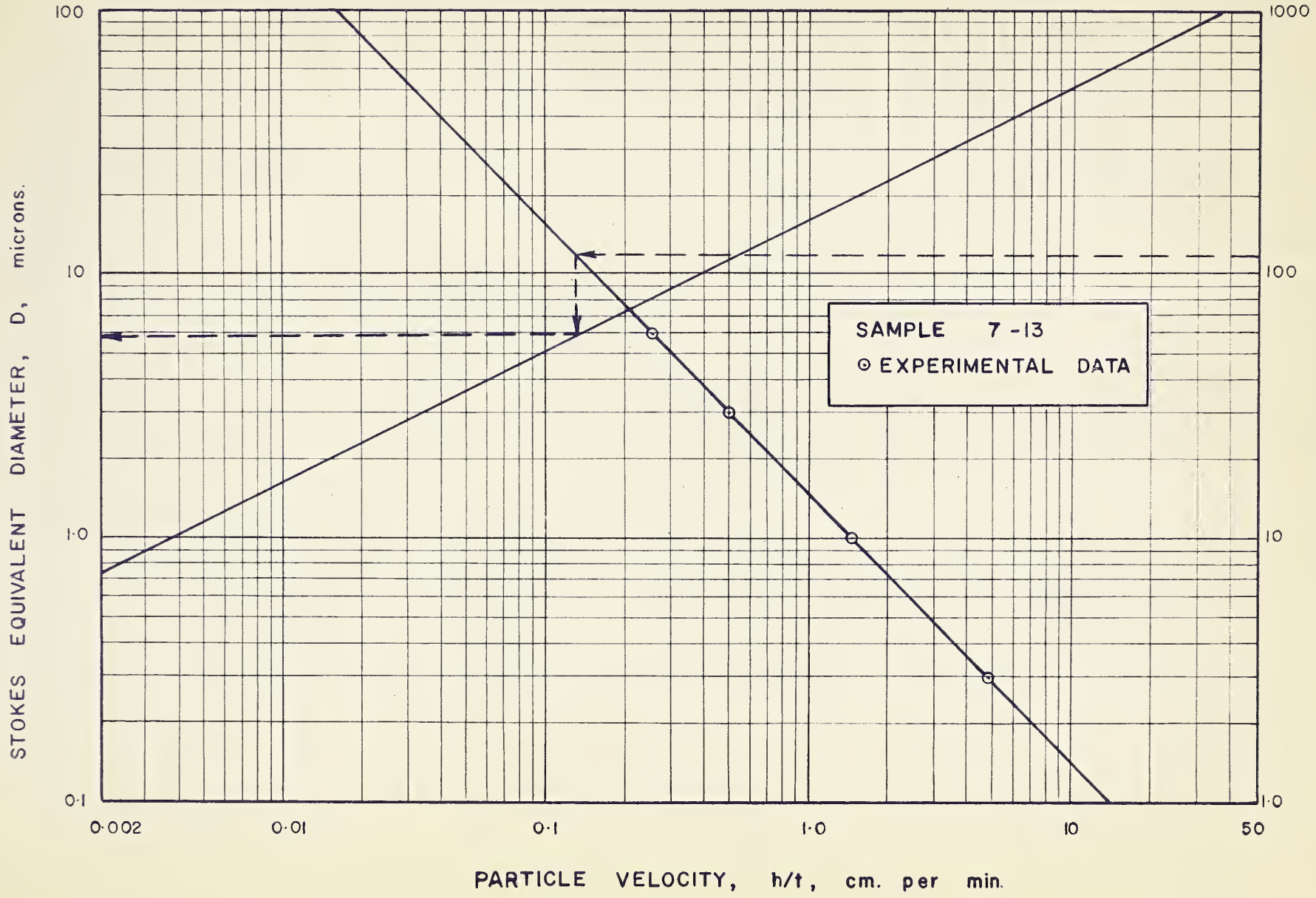

FIG. 5. PARTICLE DIAMETER AND SETTLING TIME AS A FUNCTION OF PARTICLE VELOCITY.

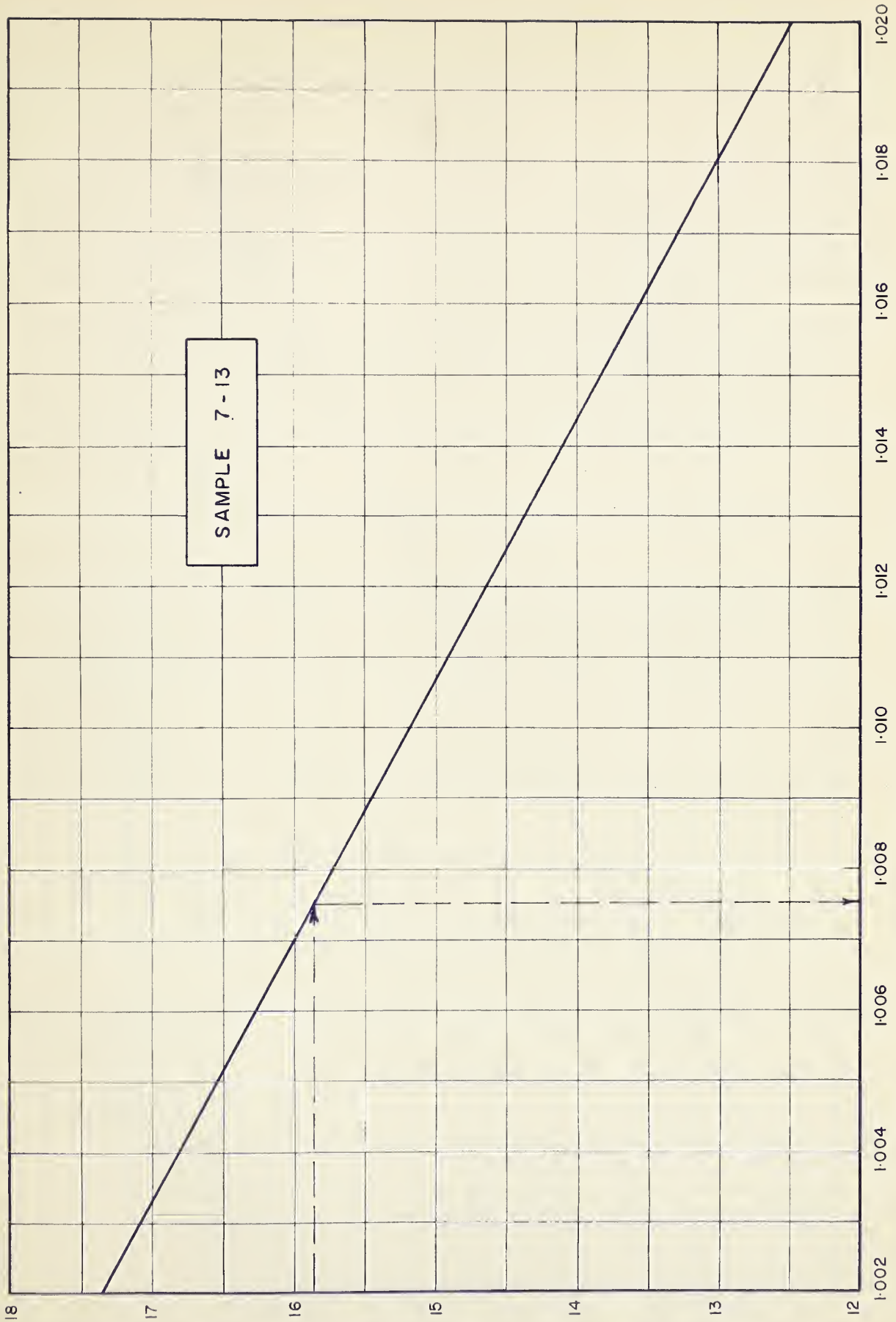

FIG. 6. EFFECTIVE DEPTH vs. SPECIFIC GRAVITY OF SUSPENSION.

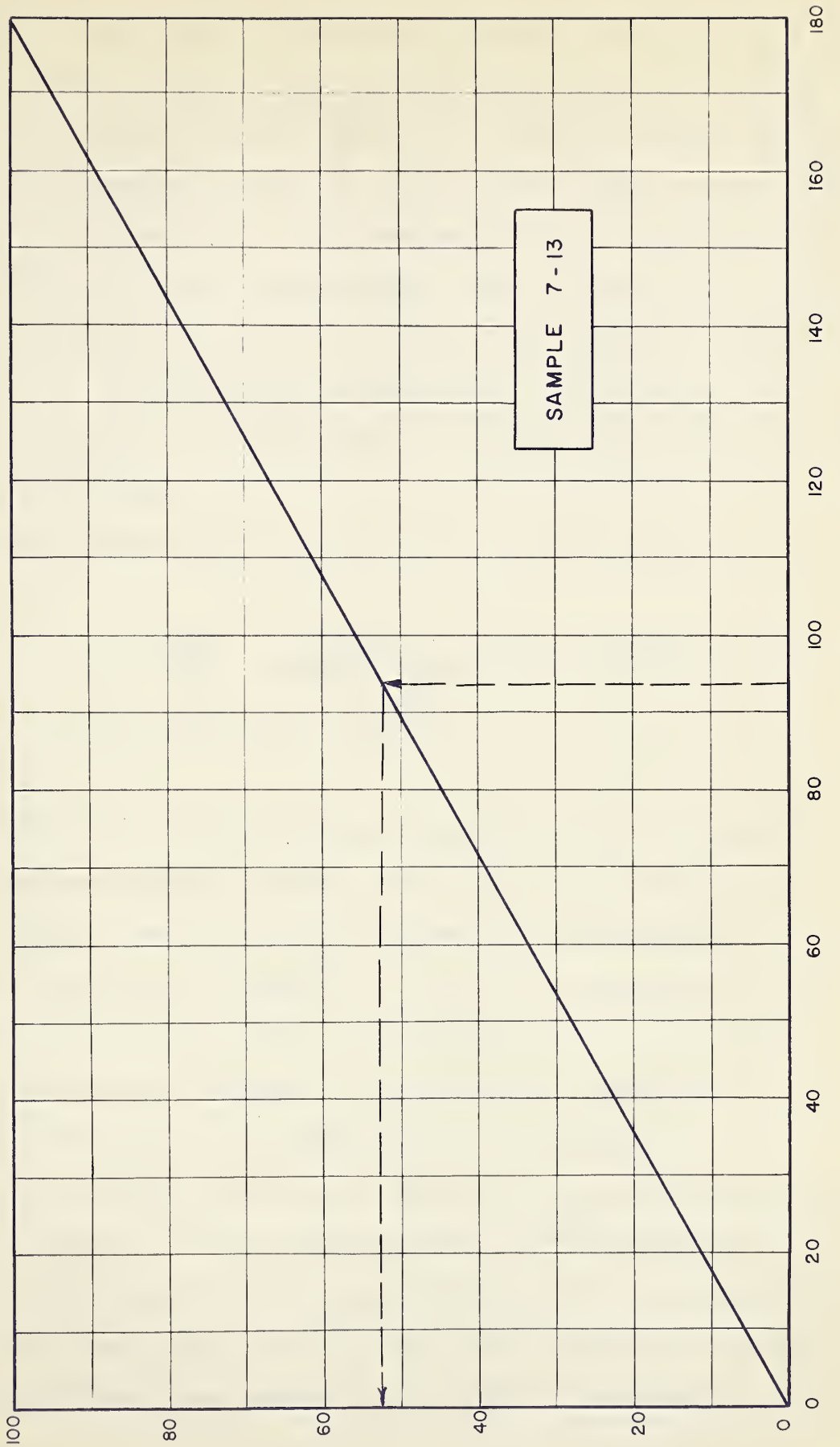

FIG. 7. CUMULATIVE WEIGHT PERCENT vs. $(d_m - d_l)$.

$\frac{h}{t}$ vs. t line can be established through the use of the h vs. G relationship, shown on Fig.6. The first step is to apply the +0.0003 meniscus correction to the observed specific gravity values. The corresponding values of the effective depth to the specific gravity observations, using hydrometer No.1, are found to be 14.02, 14.34 and 14.74 cm, respectively. From these values the settling velocities ($\frac{h}{t}$) are 4.67, 1.434 and 0.491 cm/min. Plotting the values of $\frac{h}{t}$ against the corresponding values of settling times, t, and joining the points with a straight line, Fig. 5 is complete for calculations.

The preparation of the W vs. $(d_m - d_1)$ chart is as follows:

$$W = \frac{1000}{30} \quad \frac{2.4940}{2.4940-0.9982} \quad (d_m - 0.9982) \times 100\%$$

$$W = 5{,}562 \ (d_m - 0.9982)$$

This is a straight line on linear coordinates, having a slope of 5,562 and passing through the origin as illustrated on Fig.7.

Having the three charts prepared, the calculation of the Stokes' equivalent diameter, D, and the correspodning weight percent, W, at any values of settling time, t, is straight forward. For illustration purposes the computation technique for sample 7-13 after 120 minutes of sedimentation is presented.

The settling velocity of particles, $\frac{h}{t}$, after 120 minutes is 0.1275 cm/min, and the corresponding particle diameter, D, is 5.90 microns from Fig.5. Multiplying $\frac{h}{t}$ by t it follows that h, the effective depth, is 15.31 cm., and from Fig.6, the specific gravity of the suspension, G, is 1.0095. The specific gravity

multiplied by the density of the distilled water, d_1, at $68^{\circ}F$ gives the density of the suspension, d_m, at that temperature, expressed in grams per milliliter. In this case, it was calculated to be 1.0076 gm/ml. From the knowledge of d_m, 1.0076 gm/ml, the weight percent smaller than the corresponding size, W, is found from Fig. 7 to be 52.20 percent.

The complete size distribution analysis and the additional data required in the calculations are shown in Table 1. The results are usually represented in graphical form, using semilogarithmic paper and plotting D on the logarithmic scale and W on the linear scale as illustrated on Fig. 8.

TABLE 1

EXAMPLE OF PARTICLE SIZE DISTRIBUTION CALCULATIONS

SAMPLE 7-13.

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml	cm	cm/min.	micron	wt. percent	micron
1	1.0150	1.0153	1.0134	13.76	13.7600	61.00	84.20	53.00
3	1.0140	1.0143	1.0124	14.02	4.6700	36.00	78.60	31.00
10	1.0128	1.0131	1.0112	14.34	1.4340	19.80	72.00	17.10
30	1.0113	1.0116	1.0097	14.74	0.4910	11.70	63.70	10.00
60	1.0102	1.0105	1.0086	15.04	0.2510	8.30	57.60	7.15
120	1.0092	1.0095	1.0076	15.31	0.1275	5.90	52.20	5.10
300	1.0082	1.0085	1.0066	15.58	0.0520	3.78	46.60	3.25
1440	1.0061	1.0064	1.0045	16.14	0.0112	1.76	35.00	1.52

ADDITIONAL DATA:

Concentration of suspension = 3 percent by weight
Volume taken for analysis = 79.10 ml.
Meniscus correction = +0.0003
Temperature of suspension = 80°F
Temperature correction = +0.0016 gm/ml.
Density of water at 80°F = 0.9966 gm/ml.
Viscosity of suspension at 68°F = 1.30 cp.
Density of solids = 2.4940 gm/ml.
Viscosity of water at 68°F = 1.0005 cp.

* Values of D calculated, using the viscosity of water.

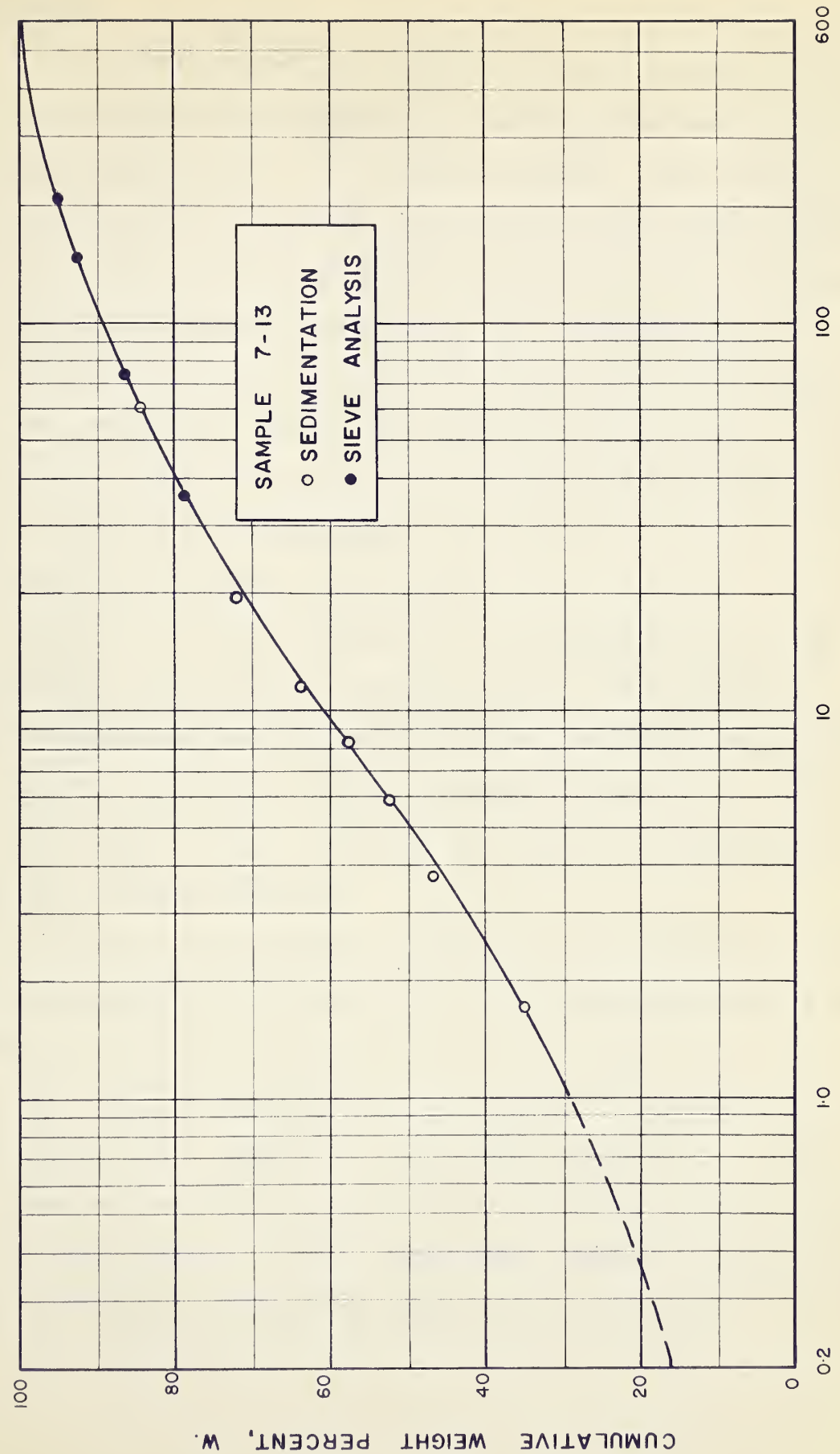

FIG. 8. PARTICLE SIZE DISTRIBUTION.

DISCUSSION

It has been pointed out that the purpose of this investigation was to establish a rapid particle size distribution analysis of drilling fluids in the 2 to 74 micron range. This purpose has been fulfilled by developing the previously described graphical approach. The question naturally arises about the reliability of the method, its relative advantages and disadvantages.

The first and most significant advantage of the method is that the time required to obtain the distribution function is enormously shortened since only three measurements of the specific gravity of the suspension are necessary. Therefore, the usual test time of 24 hours is reduced to 30 minutes.

Secondly, the time required of calculating the results is also shortened utilizing the prepared graphs, and therefore, for routine check analyses the method is particularly advantageous.

Thirdly, the three specific gravity measurements, instead of the usual eight or more, decrease the effect of turbulence in the settling of small particles.

Of course, the graphical solution of any problem has some hidden inaccuracies which can not be eliminated but only reduced by taking care in the preparation of the graphs, and later in reading them. This is probably the most serious disadvantage of the method described. However, it has been experienced by the author that these errors are not significant if proper care is taken, and the investigator is well compensated timewise for any possible errors introduced through the graphical calculations.

A study of methods of particle size analysis that utilize sedimentation showed that in nearly all of the methods the assumption has been made that the viscosity of the suspension is the same as the viscosity of distilled water. Gates (10) was the first investigator who suggested that in case of drilling fluids the viscosity of the suspension should be measured and used in the calculations, but he did not verify it experimentally.

To justify the use of suspension viscosity in the calculations, and for other purposes which will be discussed later, 3 and 5 percent by weight suspensions were prepared and sedimented in conjunction with microscopic and sieve analyses. Two typical sets of data are presented in Table 2 and Table 3 and plotted on Figures 9, 10, 11 and 12. (The rest of the experimental and calculated data can be found in Tables 5 to 26 in the appendix.)

The most striking feature of these illustrations is that using the suspension viscosity, in the case of 3 percent suspension, the sedimentation data correlated very well with the size distribution line obtained from microscopic and sieve analyses of identical samples, as shown in Fig. 11 and Fig.12. Deviations from the lines were experienced on both ends of the lines determined from the microscopic measurements, which was probably due to the deflocculating agent used in the microscopic analyses. The agreement between the microscopic analyses and the sedimentation of 5 percent suspensions was not as close but for practical purposes it was good. The maximum deviation in both the 3 and 5 percent suspensions never amounted up to more than $\pm$ 3 percent and averaged around $\pm$ 1.5 percent.

TABLE 2.

COMPARISON OF SEDIMENTATION, MICROSCOPIC AND SIEVE ANALYSES. SAMPLES 7-13 AND 7-15.

SEDIMENTATION.

3% suspension			5% suspension		
D	W	D*	D	W	D*
micron	wt.percent	micron	micron	wt.percent	micron
61.00	84.20	53.00	59.50	84.40	47.50
36.00	78.60	31.00	35.00	79.00	28.20
19.80	72.00	17.10	19.40	73.40	15.60
11.70	63.70	10.00	11.50	63.70	9.30
8.30	57.60	7.15	8.30	58.00	6.70
5.90	52.20	5.10	5.95	52.30	4.80
3.78	46.60	3.25	3.85	46.60	3.10
1.76	35.00	1.52	1.80	38.30	1.45

Sieve and Microscopic Analyses.

D	W	D	W
micron	wt.percent	micron	wt.percent
210**	94.90	12.0	64.00
149**	92.30	9.3	60.10
74**	86.50	7.0	55.90
60	84.10	5.8	53.10
50	82.10	4.6	49.90
40	79.50	3.7	47.20
30	76.50	2.8	43.50
25	74.50	2.1	40.30
20	71.00	1.7	38.20
16	67.50	1.3	35.20

* Values of D calculated using the viscosity of water.

** Sieve analysis.

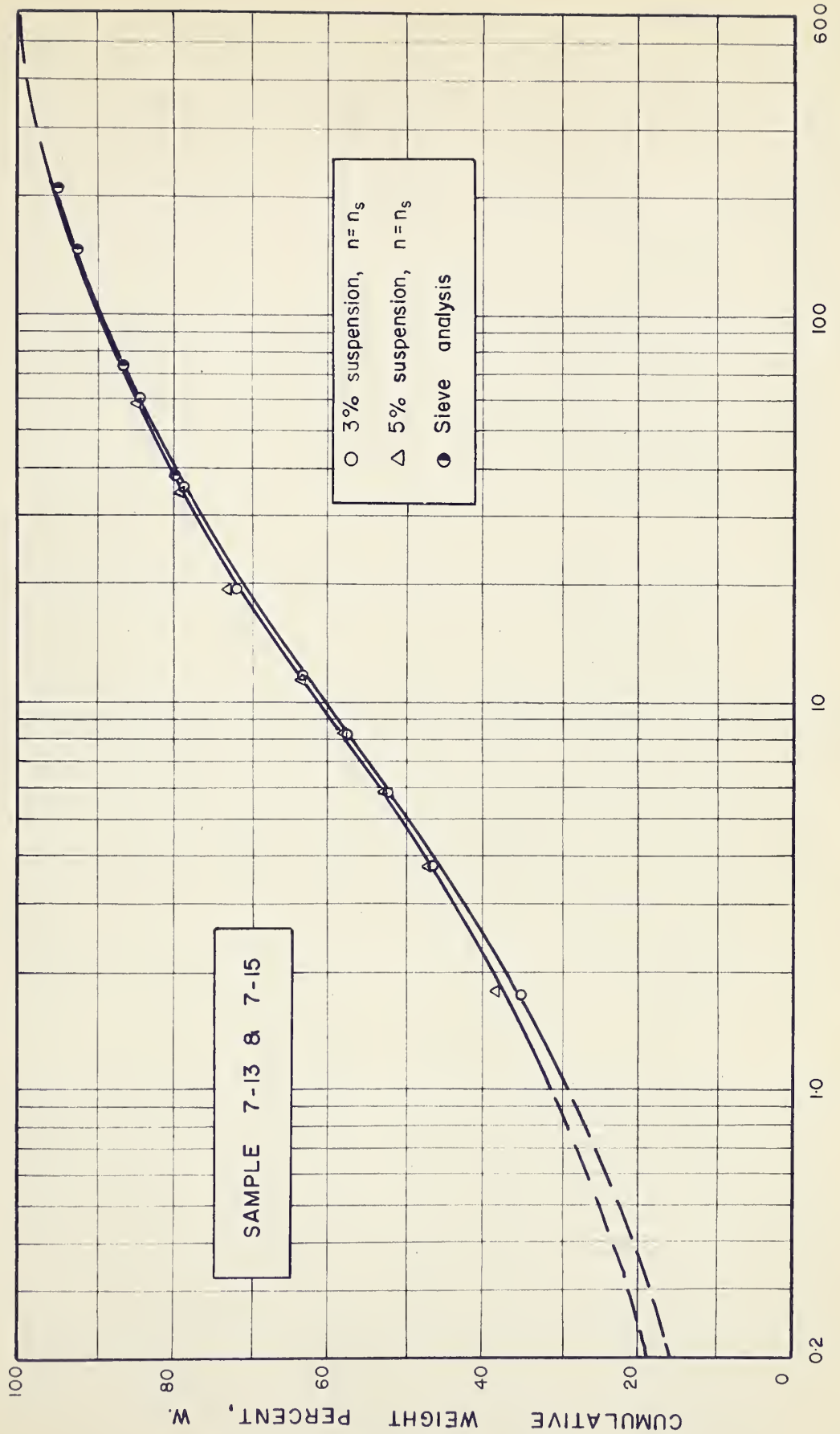

FIG. 9. PARTICLE SIZE DISTRIBUTION, USING THE VISCOSITY OF SUSPENSION.

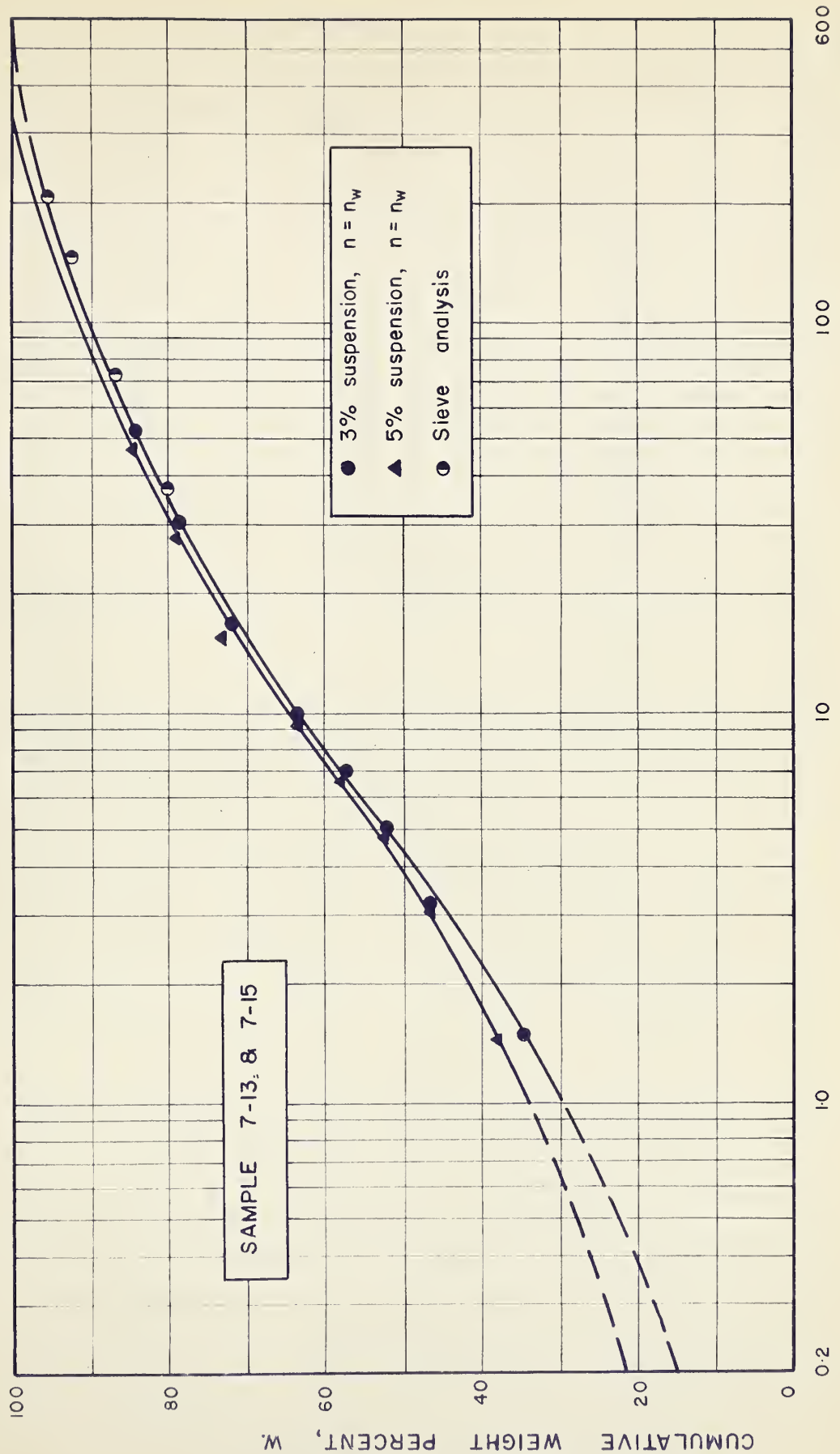

FIG. 10. PARTICLE SIZE DISTRIBUTION, USING THE VISCOSITY OF WATER.

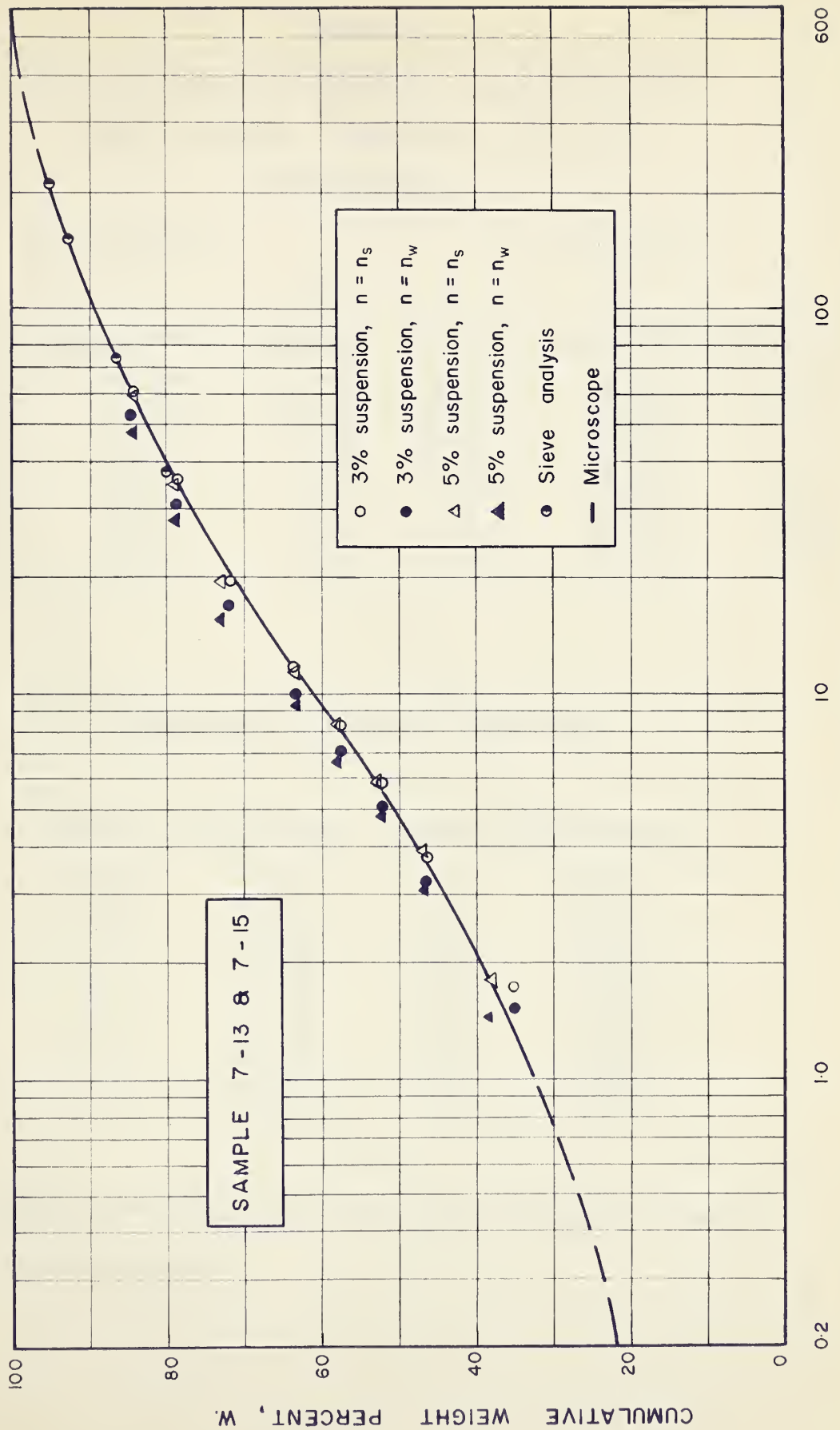

FIG. 11. COMPARISON OF MICROSCOPIC, SIEVE AND SEDIMENTATION ANALYSES.

TABLE 3.

COMPARISON OF SEDIMENTATION, MICROSCOPIC AND SIEVE ANALYSES. SAMPLES 6-13 AND 6-15.

SEDIMENTATION.

3% suspension			5% suspension		
D	W	D*	D	W	D*
micron	wt.percent	micron	micron	wt.percent	micron
57.50	80.30	50.70	55.60	80.10	43.45
34.20	65.90	30.05	34.05	66.40	26.55
19.15	52.50	16.85	19.45	54.20	15.15
11.23	43.90	9.86	11.58	44.30	9.05
8.00	38.55	7.03	8.36	38.80	6.52
5.69	34.25	5.00	5.97	34.90	4.65
3.63	29.45	3.19	3.83	30.10	2.98
1.68	21.40	1.47	1.79	22.90	1.40

Sieve and Microscopic Analyses.

D	W	D	W
micron	wt.percent	micron	wt.percent
149**	96.50	12.0	45.40
105**	91.00	9.3	41.90
74**	84.10	7.0	37.20
60	79.10	5.8	35.60
50	74.90	4.6	34.10
40	69.50	3.7	31.00
30	61.60	2.8	27.90
25	58.50	2.1	25.80
20	54.10	1.7	24.50
16	50.50	1.3	22.40

* Values of D calculated using the viscosity of water.

** Sieve analysis.

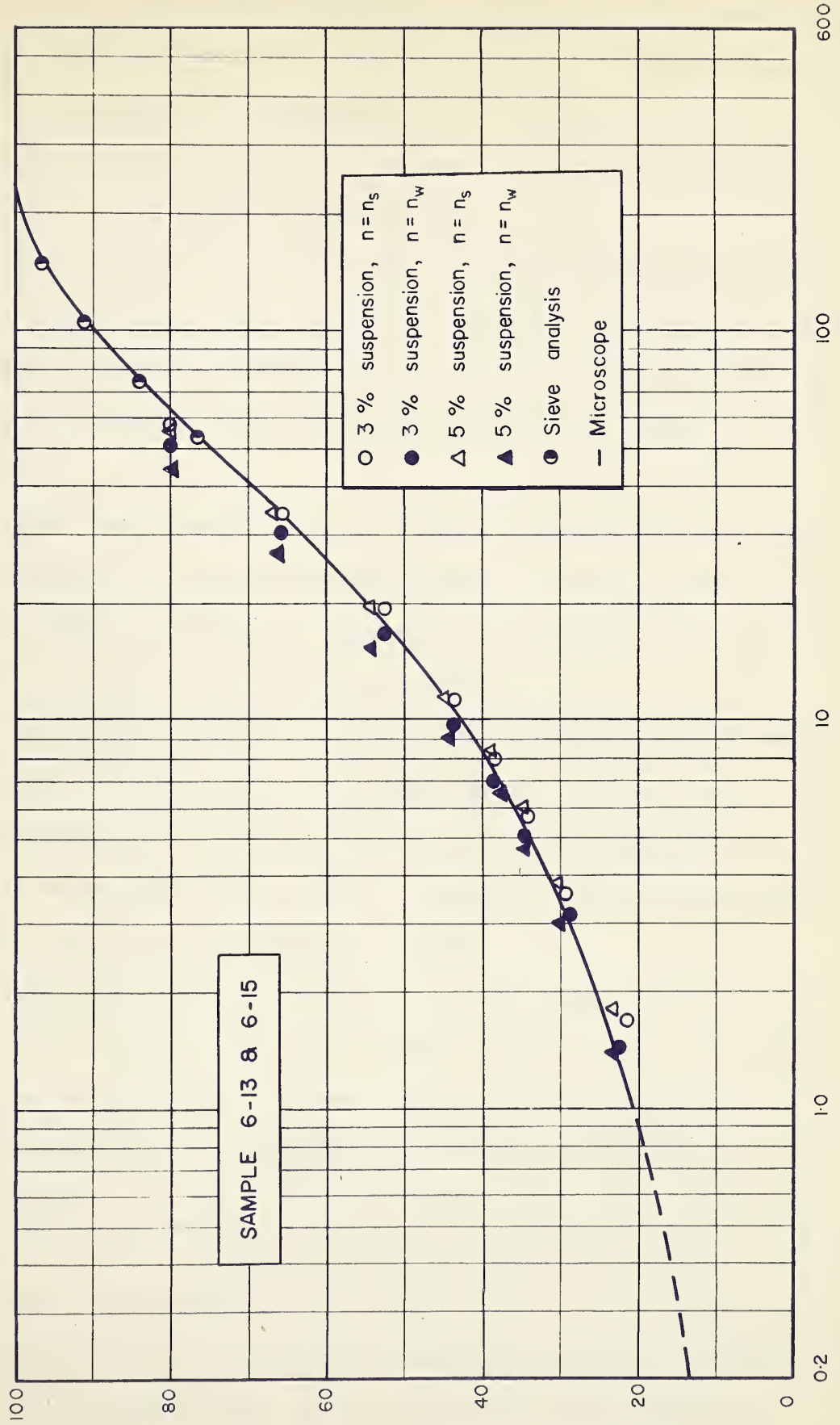

FIG. 12. COMPARISON OF MICROSCOPIC, SIEVE AND SEDIMENTATION ANALYSES.

On the other hand, using the viscosity of the distilled water in the calculations the agreement between the microscopic results and the sedimentation data was poor. Furthermore, there was no agreement between the data obtained from the sedimentation of the 3 percent and 5 percent suspensions.

The effect of viscosity is striking especially in the case of larger particles, above about 25 microns. It can be seen from the samples cited that 8-10 micron differences can be obtained for identical samples using the viscosity of the water in the calculations.

From the investigations it can be definitely concluded that the viscosity of the suspension must be used for the calculations in the study of drilling fluids, and it can not be assumed being equal to that of the distilled water.

The effect of the concentration of solids on the validity of Stokes' law has not been investigated systematically, although by experimentation Martin (11) was able to estimate that Stokes' law was applicable up to about 3 percent solids for titanium dioxide particles suspended in water.

The investigations reported herein suggest that about 3 percent solids is the limit for accurate sedimentation analysis of drilling fluids also. Above 3 percent concentration the viscosity of the suspension increases sharply which influences the settling of particles. It is believed by the author that some degree of "after swelling" of the bentonite particles takes place in contact with the sedimentation liquid, even if the samples are in the fluid form before the analyses, because they are diluted and more water is available than in the drilling fluid itself.

In nearly all previously described methods of particle size analysis of soils small quantities of some chemicals were added to the suspension to aid in the dispersion of the small particles. Broughton and Hand (12) showed that the properties of a clay suspension in water depends on the ions present and the suspension has certain properties when sodium salts are present and different properties when barium salts are present. Accordingly, the use of a dispersing agent in the particle size analysis of a drilling fluid would limit the analysis of the fluid to one in which the particular chemical was present. In other words, the particle size distribution as measured using a deflocculating agent, would be the distribution of particles occurring only when the particular chemical is present and would differ for each different dispersing agent. The findings of Broughton and Hand were verified in this investigation, as shown in the appendix, and the use of any dispersing agent was discontinued and is not recommended.

The turbulent effect caused by the repeated insertion of the hydrometer into the suspension has been considered and investigated. It has been found that this effect is neglegible in the proposed method since only three measurements are necessary and the free settling of the larger particles is not affected greatly. However, the effect of fluid motion after shaking the suspension at the initiation of the test might be disturbing in the early measurements of the specific gravity of the suspension, causing error in the calculated results. To eliminate this detrimental effect the first reading was taken after 3 minutes of settling since the nature of the calculations developed herein makes it possible to calculate the particle size distribution at any earlier time desired.

CONCLUSIONS AND RECOMMENDATIONS

It is recognized that the limitations in the application of the hydrometer method to the particle size distribution analysis are in accordance with the limitations in the use of Stokes' law; to this extent the usefulness of any sedimentation method is limited.

There are several inherent advantages in the hydrometer method presented herein which justify its use in both practical and theoretical applications. It may therefore be concluded, that:

1. The equipment required is simple and inexpensive.
2. The characteristics of the hydrometer do not change, and only an initial calibration is required.
3. The experimental work to establish the distribution of particles requires only 30 minutes.
4. The turbulent effect of the insertion of the hydrometer is minimized by making three specific gravity measurements only.
5. The calculation of the distribution function is simplified by the use of prepared charts.
6. The viscosity of the suspension is measured and used in the calculation of the particle diameter.
7. No dispersing agent is added to the suspension.
8. Three percent by weight (or less) suspensions should be taken for the analysis to reduce the deviation from Stokes' law.
9. The results obtained by the proposed hydrometer method for the samples analyzed agree well with those obtained by other techniques.

The accuracy of the method can be increased by the improvement of the techniques available for the measurement of the viscosity of the suspension and for the determination of the density of the solid materials.

One long test (24 hours) is recommended in dealing with samples from new fields in order to determine that no particle size ranges are missing in the 2 to 74 micron size interval, because the proposed method is not able to detect any such discontinuities. Furthermore it is recommended that a greater variety of samples be analyzed from different geographical locations in future investigations.

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APPENDICES

TABLE 4.

PHYSICAL PROPERTIES OF SAMPLES.

SAMPLE NO.	DENSITY OF SOLIDS	VOLUME TAKEN FOR ANALYSIS	VISCOSITY OF SUSPENSION AT 68°F	TEMPERATURE OF SUSPENSION
	gm/ml	ml	cp.	°F
1-21	2.255	46.60	1.10	80
1-22	2.255	93.20	1.25	80
1-23	2.255	139.80	1.40	80
1-25	2.255	233.00	1.75	80
2-23	2.425	138.00	1.35	80
2-25	2.425	230.00	1.65	80
3-23	2.542	121.50	1.30	80
3-25	2.542	202.50	1.70	80
4-23	2.380	157.90	1.35	80
4-25	2.380	263.20	1.75	80
5-23	2.474	114.20	1.35	80
5-25	2.474	190.30	1.65	80
6-13	2.647	73.00	1.30	80
6-15	2.647	121.80	1.55	80
7-13	2.494	79.10	1.30	80
7-15	2.494	131.70	1.55	80

Density of water at 68°F = 0.9982 gm/ml.

Density of water at 80°F = 0.9966 gm/ml.

Viscosity of water at 68°F = 1.005 cp.

EXPERIMENTAL AND CALCULATED DATA.

TABLE 5. SAMPLE 1-21

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0052	1.0055	1.0036	16.39	16.3900	66.90	96.90	63.45
3	1.0051	1.0054	1.0035	16.42	5.4800	38.25	95.10	35.10
10	1.0050	1.0053	1.0034	16.45	1.6450	21.00	93.30	20.05
30	1.0041	1.0044	1.0025	16.68	0.5560	12.20	77.15	11.80
60	1.0040	1.0043	1.0024	16.71	0.2785	8.64	75.40	8.26
120	1.0034	1.0037	1.0018	16.87	0.1406	6.14	64.55	5.87
300	1.0030	1.0033	1.0014	16.97	0.0565	3.89	57.40	3.72
1440	1.0026	1.0029	1.0010	17.08	0.0118	1.78	50.20	1.63
2880	1.0024	1.0027	1.0008	17.14	0.0059	1.26	46.60	1.21

TABLE 6. SAMPLE 1-22

1	1.0108	1.0111	1.0092	14.88	14.8800	66.95	98.70	60.50
3	1.0105	1.0108	1.0089	14.96	4.9900	39.25	95.90	34.90
10	1.0103	1.0106	1.0087	15.01	1.5010	21.40	94.20	19.15
30	1.0099	1.0102	1.0083	15.12	0.5040	12.35	90.60	11.10
60	1.0091	1.0094	1.0075	15.34	0.2560	8.84	83.40	7.93
120	1.0081	1.0084	1.0065	15.60	0.1300	6.30	73.55	5.64
300	1.0070	1.0073	1.0054	15.90	0.0530	4.01	64.55	3.60
1440	1.0059	1.0062	1.0043	16.20	0.0112	1.85	54.60	1.65
2880	1.0050	1.0053	1.0034	16.44	0.0057	1.32	46.65	1.18

* Values of D calculated using water viscosity.

EXPERIMENTAL AND CALCULATED DATA.

TABLE 7. SAMPLE 1-23

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0163	1.0166	1.0147	13.41	13.4100	67.55	98.70	57.30
3	1.0160	1.0163	1.0144	13.49	4.5000	39.15	96.90	33.20
10	1.0157	1.0160	1.0141	13.57	1.3570	21.55	95.00	18.25
30	1.0149	1.0152	1.0133	13.78	0.4600	12.51	90.30	10.60
60	1.0139	1.0142	1.0123	14.05	0.2340	8.93	84.30	8.30
120	1.0123	1.0126	1.0107	14.48	0.1205	6.40	74.80	5.43
300	1.0110	1.0113	1.0094	14.83	0.0495	4.10	67.00	3.48
1440	1.0090	1.0093	1.0074	15.36	0.0107	1.91	55.00	1.62
2880	1.0076	1.0079	1.0060	15.74	0.0055	1.36	46.60	1.16

TABLE 8. SAMPLE 1-25

1	1.0274	1.0277	1.0258	10.45	10.4500	66.70	99.10	50.60
3	1.0268	1.0271	1.0252	10.61	3.5400	38.85	96.90	29.45
10	1.0263	1.0266	1.0247	10.75	1.0750	21.40	95.10	16.20
30	1.0251	1.0254	1.0235	11.06	0.3690	12.50	90.80	9.50
60	1.0230	1.0233	1.0214	11.62	0.1937	8.90	83.25	6.89
120	1.0210	1.0213	1.0194	12.15	0.1010	6.52	76.10	4.95
300	1.0181	1.0184	1.0165	12.93	0.0432	4.30	65.60	3.26
1440	1.0150	1.0153	1.0134	13.75	0.0096	2.02	54.50	1.53
2880	1.0130	1.0133	1.0114	14.29	0.0049	1.45	47.40	1.10

* Values of D calculated using water viscosity.

EXPERIMENTAL AND CALCULATED DATA.

TABLE 9. SAMPLE 1-23X**

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0164	1.0167	1.0148	13.38	13.3800	67.55	99.20	57.20
3	1.0162	1.0165	1.0146	13.44	4.4800	39.00	98.00	33.10
10	1.0153	1.0156	1.0137	13.68	1.3680	21.55	92.75	18.30
30	1.0138	1.0141	1.0122	14.08	0.4695	12.65	83.80	10.70
60	1.0130	1.0133	1.0114	14.29	0.2380	9.05	78.90	7.64
120	1.0119	1.0122	1.0103	14.59	0.1215	6.44	72.40	5.45
300	1.0102	1.0105	1.0086	15.05	0.0515	4.14	62.20	3.55
1440	1.0079	1.0082	1.0063	15.65	0.0109	1.92	48.40	1.64

TABLE 10. SAMPLE 1-25X

1	1.0273	1.0276	1.0257	10.48	10.4800	66.80	98.60	50.60
3	1.0271	1.0274	1.0255	10.53	3.5100	38.75	98.00	29.35
10	1.0258	1.0261	1.0242	10.88	1.0880	21.60	93.30	16.35
30	1.0231	1.0234	1.0215	11.60	0.3870	12.85	83.60	9.74
60	1.0212	1.0215	1.0196	12.10	0.2020	9.27	76.90	7.02
120	1.0196	1.0199	1.0180	12.53	0.1045	6.68	71.00	5.05
300	1.0171	1.0174	1.0155	13.20	0.0440	4.33	62.10	3.29
1440	1.0138	1.0141	1.0122	14.08	0.0098	2.04	50.20	1.55

** 50 ml 0.1 n NH_4 OH added.

EXPERIMENTAL AND CALCULATED DATA.

TABLE 11. SAMPLE 2-23

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0161	1.0164	1.0145	13.46	13.4600	62.40	92.40	53.80
3	1.0160	1.0163	1.0144	13.49	4.5000	36.10	91.80	31.10
10	1.0158	1.0161	1.0142	13.55	1.3550	19.80	90.70	17.10
30	1.0148	1.0151	1.0132	13.80	0.4600	11.50	85.00	9.95
60	1.0139	1.0142	1.0123	14.05	0.2340	8.23	79.90	7.10
120	1.0123	1.0126	1.0107	14.48	0.1205	5.90	70.90	5.09
300	1.0114	1.0117	1.0098	14.72	0.0491	3.76	65.75	3.25
1440	1.0094	1.0097	1.0078	15.25	0.0106	1.75	54.40	1.51

TABLE 12. SAMPLE 2-23X

1	1.0150	1.0153	1.0134	13.75	13.7500	63.10	86.20	54.50
3	1.0144	1.0147	1.0128	13.92	4.6450	36.65	82.70	31.40
10	1.0140	1.0143	1.0124	14.02	1.4020	20.15	80.50	17.40
30	1.0127	1.0130	1.0111	14.37	0.4790	11.80	73.10	10.15
60	1.0119	1.0122	1.0103	14.58	0.2430	8.39	68.60	7.23
120	1.0109	1.0112	1.0093	14.85	0.1238	5.99	62.90	5.17
300	1.0100	1.0103	1.0084	15.09	0.0503	3.81	57.80	3.29
1440	1.0080	1.0083	1.0064	15.63	0.0108	1.77	46.45	1.53

EXPERIMENTAL AND CALCULATED DATA.

TABLE 13. SAMPLE 2-25

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0280	1.0283	1.0264	10.28	10.2800	60.30	95.90	47.10
3	1.0278	1.0281	1.0262	10.34	3.4500	34.90	95.20	27.30
10	1.0263	1.0266	1.0247	10.74	1.0740	19.50	90.10	15.20
30	1.0246	1.0249	1.0230	11.20	0.3735	11.50	84.40	8.99
60	1.0224	1.0227	1.0208	11.78	0.1965	8.34	76.90	6.50
120	1.0205	1.0208	1.0189	12.29	0.1025	6.02	70.40	4.70
300	1.0187	1.0190	1.0171	12.76	0.0425	3.88	64.30	3.04
1440	1.0152	1.0155	1.0136	13.70	0.0095	1.84	52.40	1.44

TABLE 14. SAMPLE 2-25X

1	1.0258	1.0261	1.0242	10.88	10.8800	62.00	88.40	48.50
3	1.0250	1.0253	1.0234	11.09	3.7000	36.10	85.70	28.25
10	1.0244	1.0247	1.0228	11.25	1.1250	19.95	83.60	15.60
30	1.0220	1.0223	1.0204	11.89	0.3965	11.80	75.50	9.24
60	1.0209	1.0212	1.0193	12.18	0.2030	8.47	71.75	6.62
120	1.0192	1.0195	1.0176	12.68	0.1057	6.11	66.00	4.78
300	1.0168	1.0171	1.0152	13.27	0.0432	3.95	57.80	3.09
1440	1.0131	1.0134	1.0115	14.26	0.0099	1.87	45.20	1.47

EXPERIMENTAL AND CALCULATED DATA.

TABLE 15. SAMPLE 3-23

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml	cm	cm/min.	micron	wt.percent	micron
1	1.0171	1.0174	1.0155	13.20	13.2000	59.40	95.00	51.30
3	1.0161	1.0164	1.0145	13.46	4.4900	34.65	89.45	29.90
10	1.0151	1.0154	1.0135	13.73	1.3730	19.20	84.00	16.60
30	1.0137	1.0140	1.0121	14.10	0.4700	11.20	76.30	9.69
60	1.0127	1.0130	1.0111	14.37	0.2395	8.00	70.80	6.90
120	1.0114	1.0117	1.0098	14.72	0.1226	5.73	63.65	4.95
300	1.0105	1.0108	1.0089	14.96	0.0499	3.64	58.75	3.15
1440	1.0083	1.0086	1.0067	15.55	0.0108	1.70	46.65	1.47

TABLE 16. SAMPLE 3-23X

1	1.0162	1.0165	1.0146	13.44	13.4400	60.00	90.00	51.80
3	1.0153	1.0156	1.0137	13.68	4.5600	34.90	85.10	30.10
10	1.0149	1.0152	1.0133	13.78	1.3780	19.20	82.85	16.60
30	1.0133	1.0136	1.0117	14.21	0.4740	11.25	74.05	9.72
60	1.0124	1.0127	1.0108	14.45	0.2410	8.03	69.10	6.94
120	1.0112	1.0115	1.0096	14.76	0.1230	5.74	62.50	4.96
300	1.0099	1.0102	1.0083	15.12	0.0504	3.67	55.40	3.17
1440	1.0079	1.0082	1.0063	15.66	0.0109	1.70	44.45	1.47

EXPERIMENTAL AND CALCULATED DATA.

TABLE 17. SAMPLE 3-25

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0284	1.0287	1.0268	10.17	10.1700	58.50	94.20	45.00
3	1.0270	1.0273	1.0254	10.56	3.5200	34.45	89.70	26.45
10	1.0253	1.0256	1.0237	11.01	1.1010	19.30	84.00	14.80
30	1.0230	1.0233	1.0214	11.62	0.3880	11.25	76.45	8.65
60	1.0213	1.0216	1.0197	12.07	0.2010	8.24	70.90	6.33
120	1.0193	1.0196	1.0177	12.60	0.1050	5.95	64.25	4.58
300	1.0170	1.0173	1.0154	13.22	0.0441	3.86	56.60	2.97
1440	1.0136	1.0139	1.0120	14.13	0.0098	1.82	45.50	1.40

TABLE 18. SAMPLE 3-25X

1	1.0280	1.0283	1.0264	10.29	10.2900	58.90	92.95	45.20
3	1.0263	1.0266	1.0247	10.74	3.5810	34.75	87.30	26.75
10	1.0253	1.0256	1.0237	11.01	1.0101	19.30	84.05	14.80
30	1.0230	1.0233	1.0214	11.62	0.3880	11.25	76.50	8.65
60	1.0211	1.0214	1.0195	12.13	0.2021	8.25	70.20	6.35
120	1.0192	1.0195	1.0176	12.64	0.1050	5.95	63.95	4.58
300	1.0173	1.0176	1.0157	13.15	0.0439	3.84	57.60	2.46
1440	1.0137	1.0140	1.0121	14.10	0.0098	1.81	45.80	1.39

EXPERIMENTAL AND CALCULATED DATA.

TABLE 19. SAMPLE 4-23

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0162	1.0165	1.0146	13.44	13.4400	63.40	94.10	54.60
3	1.0155	1.0158	1.0139	13.62	4.5400	36.85	90.10	31.75
10	1.0152	1.0155	1.0136	13.70	1.3700	20.25	88.40	17.50
30	1.0141	1.0144	1.0125	14.00	0.4670	11.80	82.05	10.20
60	1.0132	1.0135	1.0116	14.24	0.2375	8.43	76.90	7.26
120	1.0119	1.0122	1.0103	14.58	0.1214	6.03	69.40	5.20
300	1.0106	1.0109	1.0090	15.20	0.0507	3.89	62.00	3.36
1440	1.0092	1.0095	1.0076	15.31	0.0106	1.79	53.95	1.54

TABLE 20. SAMPLE 4-25

1	1.0272	1.0275	1.0256	10.50	10.5000	63.85	94.30	48.30
3	1.0260	1.0263	1.0244	10.82	3.6100	37.25	90.20	28.20
10	1.0253	1.0256	1.0237	11.01	1.1010	20.65	87.85	15.60
30	1.0229	1.0232	1.0213	11.65	0.3885	12.30	79.50	9.30
60	1.0213	1.0216	1.0197	12.07	0.2010	8.83	74.00	6.68
120	1.0192	1.0195	1.0176	12.64	0.1052	6.39	66.80	4.84
300	1.0176	1.0179	1.0160	13.06	0.0435	4.11	61.30	3.11
1440	1.0150	1.0153	1.0134	13.76	0.0096	1.92	52.30	1.45

EXPERIMENTAL AND CALCULATED DATA.

TABLE 21. SAMPLE 5-23

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0169	1.0172	1.0153	13.25	13.2500	61.00	95.55	52.75
3	1.0162	1.0165	1.0146	13.44	4.4850	35.50	91.70	30.50
10	1.0152	1.0155	1.0136	13.70	1.3700	19.60	86.05	16.95
30	1.0137	1.0140	1.0121	14.10	0.4700	11.50	77.60	9.93
60	1.0121	1.0124	1.0105	14.54	0.2420	8.25	68.70	7.12
120	1.0112	1.0115	1.0096	14.77	0.1230	5.89	63.70	5.09
300	1.0096	1.0099	1.0080	15.20	0.0507	3.77	54.70	3.26
1440	1.0078	1.0081	1.0062	15.69	0.0109	1.75	44.70	1.51

TABLE 22. SAMPLE 5-25

1	1.0278	1.0281	1.0262	10.34	10.3400	59.45	93.85	46.55
3	1.0270	1.0273	1.0254	10.55	3.5200	34.65	91.20	27.15
10	1.0243	1.0246	1.0227	11.27	1.1270	19.60	82.05	15.35
30	1.0220	1.0223	1.0204	11.89	0.3960	11.80	74.35	9.25
60	1.0199	1.0202	1.0183	12.45	0.2075	8.40	67.40	6.60
120	1.0182	1.0185	1.0166	12.90	0.1075	6.06	61.60	4.75
300	1.0172	1.0175	1.0156	13.17	0.0439	3.87	58.30	3.04
1400	1.0148	1.0151	1.0132	14.32	0.0099	1.85	50.20	1.45

EXPERIMENTAL AND CALCULATED DATA.

TABLE 23. SAMPLE 6-13

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml.	cm	cm/min.	micron	wt. percent	micron
1	1.0148	1.0151	1.0132	13.81	13.8100	57.75	80.30	50.70
3	1.0121	1.0124	1.0105	14.53	4.8500	34.20	65.90	30.05
10	1.0096	1.0199	1.0080	15.20	1.5200	19.15	52.50	16.85
30	1.0080	1.0083	1.0064	15.63	0.5210	11.23	43.90	9.86
60	1.0070	1.0073	1.0054	15.90	0.2650	8.00	38.55	7.03
120	1.0062	1.0065	1.0046	16.11	0.1341	5.69	34.25	5.00
300	1.0053	1.0056	1.0037	16.36	0.0545	3.63	29.45	3.19
1440	1.0038	1.0041	1.0022	16.76	0.0116	1.68	21.40	1.47

TABLE 24. SAMPLE 6-15

1	1.0284	1.0287	1.0268	10.17	10.1700	55.60	80.10	43.45
3	1.0241	1.0244	1.0225	11.33	3.7800	34.05	66.40	26.55
10	1.0203	1.0206	1.0187	12.34	1.2340	19.45	54.20	15.15
30	1.0172	1.0175	1.0156	13.17	0.4390	11.58	44.30	9.05
60	1.0155	1.0158	1.0139	13.62	0.2270	8.36	38.80	6.52
120	1.0143	1.0146	1.0127	13.95	0.1162	5.97	34.90	4.65
300	1.0128	1.0131	1.0112	14.34	0.0479	3.83	30.10	2.98
1440	1.0104	1.0107	1.0088	14.98	0.0104	1.79	22.90	1.40

EXPERIMENTAL AND CALCULATED DATA.

TABLE 25. SAMPLE 7-13

t	Sp.Gr.	G	dm	h	$\frac{h}{t}$	D	W	D*
min.	-	-	gm/ml	cm	cm/min	micron	wt. percent	micron
1	1.0150	1.0153	1.0134	13.76	13.7600	61.00	84.20	53.00
3	1.0140	1.0143	1.0124	14.02	4.6700	36.00	78.60	31.00
10	1.0128	1.0131	1.0112	14.34	1.4340	19.80	72.00	17.10
30	1.0113	1.0116	1.0097	14.74	0.4910	11.70	63.70	10.00
60	1.0102	1.0105	1.0086	15.04	0.2510	8.30	57.60	7.15
120	1.0092	1.0095	1.0076	15.31	0.1275	5.90	52.20	5.10
300	1.0082	1.0085	1.0066	15.58	0.0520	3.78	46.60	3.25
1440	1.0061	1.0064	1.0045	16.14	0.0112	1.76	35.00	1.52

TABLE 26. SAMPLE 7-15

1	1.0251	1.0254	1.0235	11.06	11.0600	59.50	84.40	47.50
3	1.0235	1.0238	1.0219	11.49	3.8400	35.00	79.00	28.20
10	1.0218	1.0221	1.0202	11.94	1.1940	19.40	73.40	15.60
30	1.0189	1.0192	1.0173	12.71	0.4240	11.50	63.70	9.30
60	1.0172	1.0175	1.0156	13.17	0.2190	8.30	58.00	6.70
120	1.0155	1.0158	1.0139	13.62	0.1140	5.95	52.30	4.80
300	1.0138	1.0141	1.0122	14.08	0.0470	3.85	46.60	3.10
1440	1.0113	1.0116	1.0097	14.74	0.0103	1.80	38.30	1.45